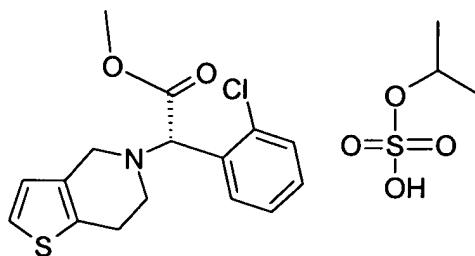


**Process for the preparation of a salt of clopidogrel with isopropyl-sulphate**

In a boiling mixture of 22 ml of acetone and 20 ml of 2-propanol 3,0 g of clopidogrel hydrogensulphate of polymorph form 1 is dissolved. The mixture is slowly cooled to room temperature then kept in fridge overnight. The precipitated crystals filtered and washed with cold acetone.

Yield: 2.18 g (66%)

Analysis: C<sub>19</sub>H<sub>24</sub>ClNO<sub>6</sub>S<sub>2</sub> (461.99)

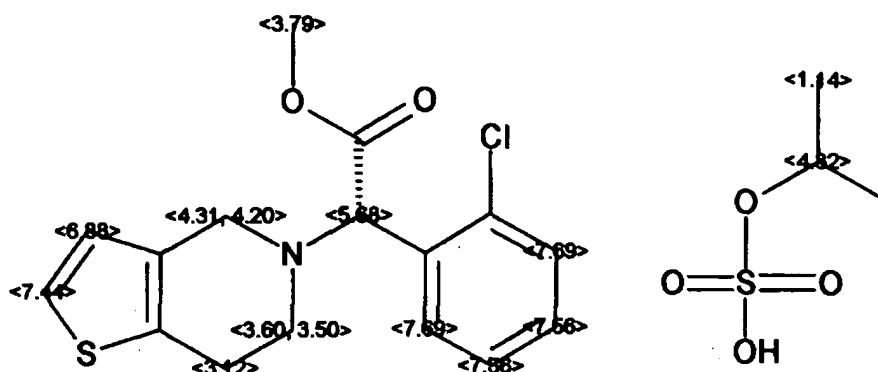
Calculated: C 49.40 %; H 5.24 %; Cl 7.67 %, N 3.03 %, S 13.88 %

Found: C 49.30 %; H 5.31 %; Cl 7.82 %; N 3.19 %, S 14.01 %.

IR, <sup>1</sup>H-NMR and <sup>13</sup>C-NMR, X-ray diffractogram are enclosed

Leadás dátum:	2001.10.09.	mérések:	IR, HNMR, CNMR	Jegyzékkönyv:	IGEN
J.k. dátuma:	2001.10.11.	Mérések:	IR, HNMR, CNMR		

1H hozzárendelések:



DMSO+D2O

13C (v. X) hozzárendelések:

IR (KBr): 3442, 1755, 1284, 1037.

HNMR (DMSO+D2O, i400): 7.69 (m, 2H), 7.56 (m, 2H), 7.44 (d, J=5.2 Hz, 1H), 6.88 (d, J=5.1 Hz, 1H), 5.68 (s, 1H), 4.32 (hp, J=6.2 Hz, 1H), 4.31 (d, 1H), 4.20 (d, J=14.7 Hz, 1H), 3.79 (s), 3.60 (m, 1H), 3.50 (m, 1H), 3.12 (m, 2H), 1.14 (d, J=6.3 Hz, 6H).  
 CNMR: 167.93, 134.94, 133.03, 132.21, 131.30, 130.71, 129.15, 128.67, 128.21, 125.99, 125.80, 69.98, 66.07, 54.39, 50.93, 49.78, 23.56, 22.78.

Jegyzékkönyv:

A spektrumok megfelelnek a fetételezett szerkezetnek. IPA hozzáadással bizonyítottuk, hogy a spektrumban látható 1 mol iPrO- csoport nem izopropanoltól származik.

Dancsó András

## STANDARD 1H OBSERVE

40660  
S2583  
Kotay Nagy Peter  
01/10/10(NM)

Solvent: DMso  
Temp. 25.0 C / 298.1 K  
File: 40660.h  
INOVA-300 "1500"

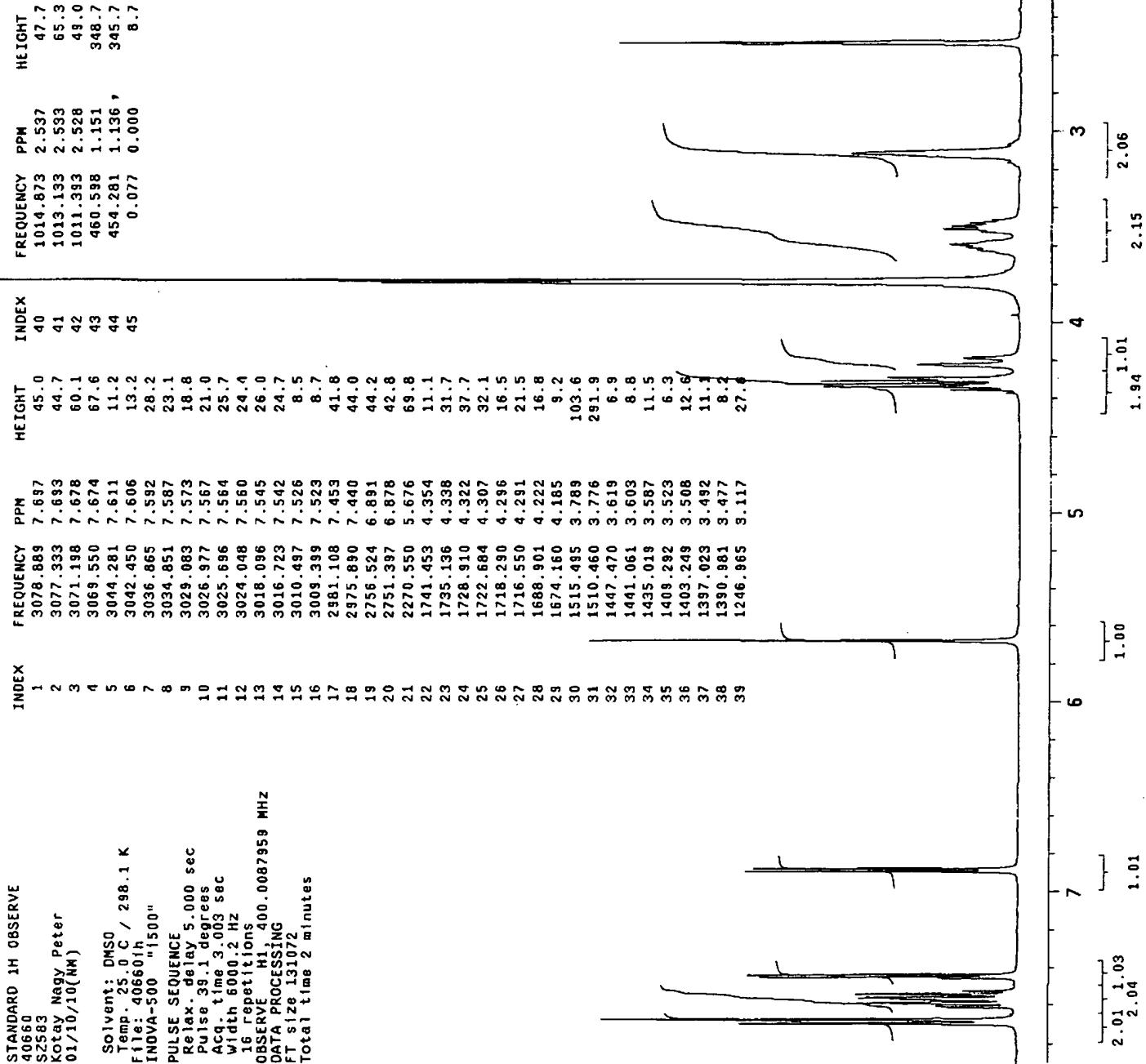
PULSE SEQUENCE  
Relax. delay 5.000 sec

Pulse 39.1 degrees  
Acq. time 3.003 sec

Width 6000.2 Hz  
16 repetitions

OBSERVE H1 400.0087959 MHZ

DATA PROCESSING  
FT S128 131072  
Total time 2 minutes

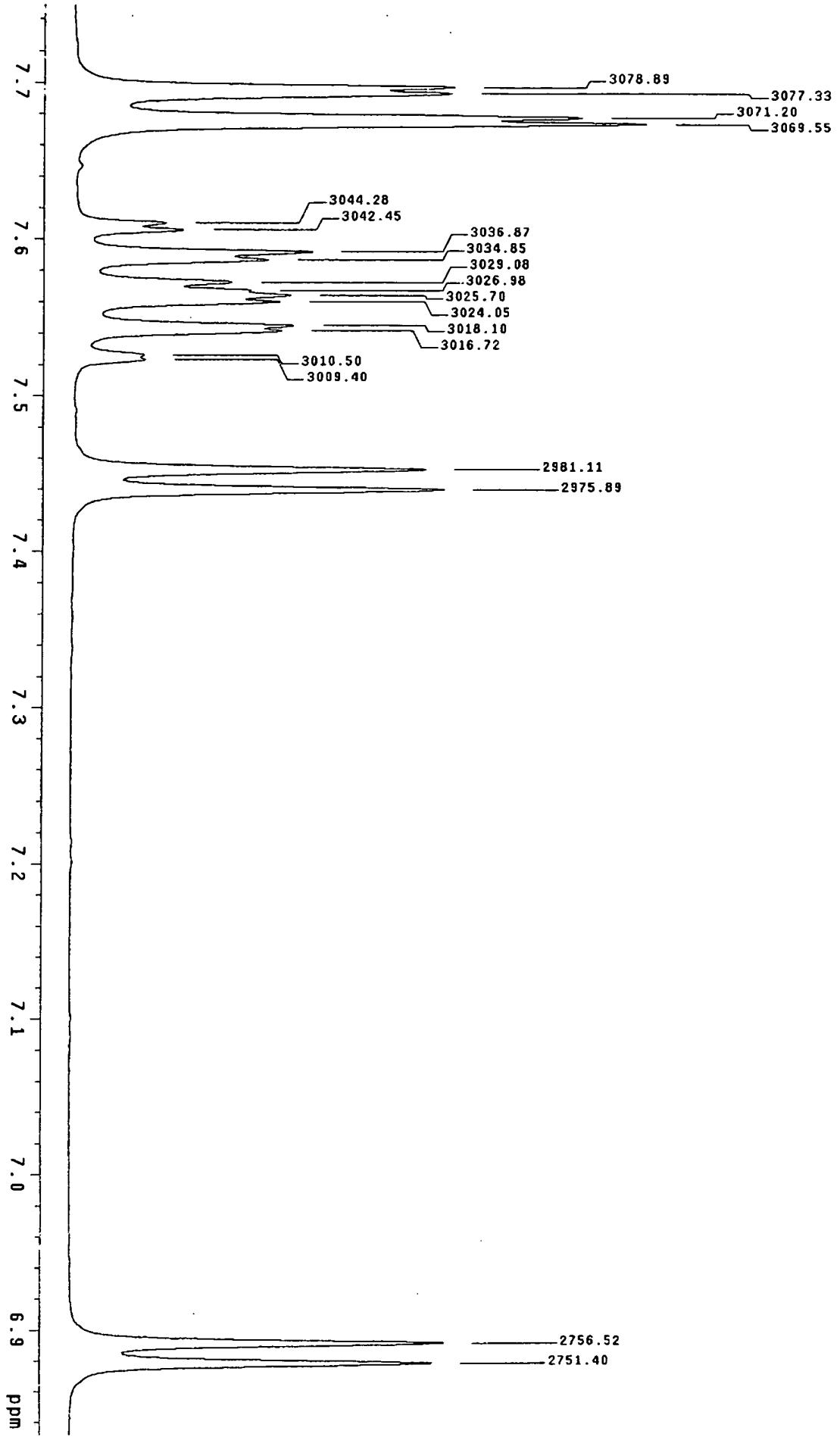


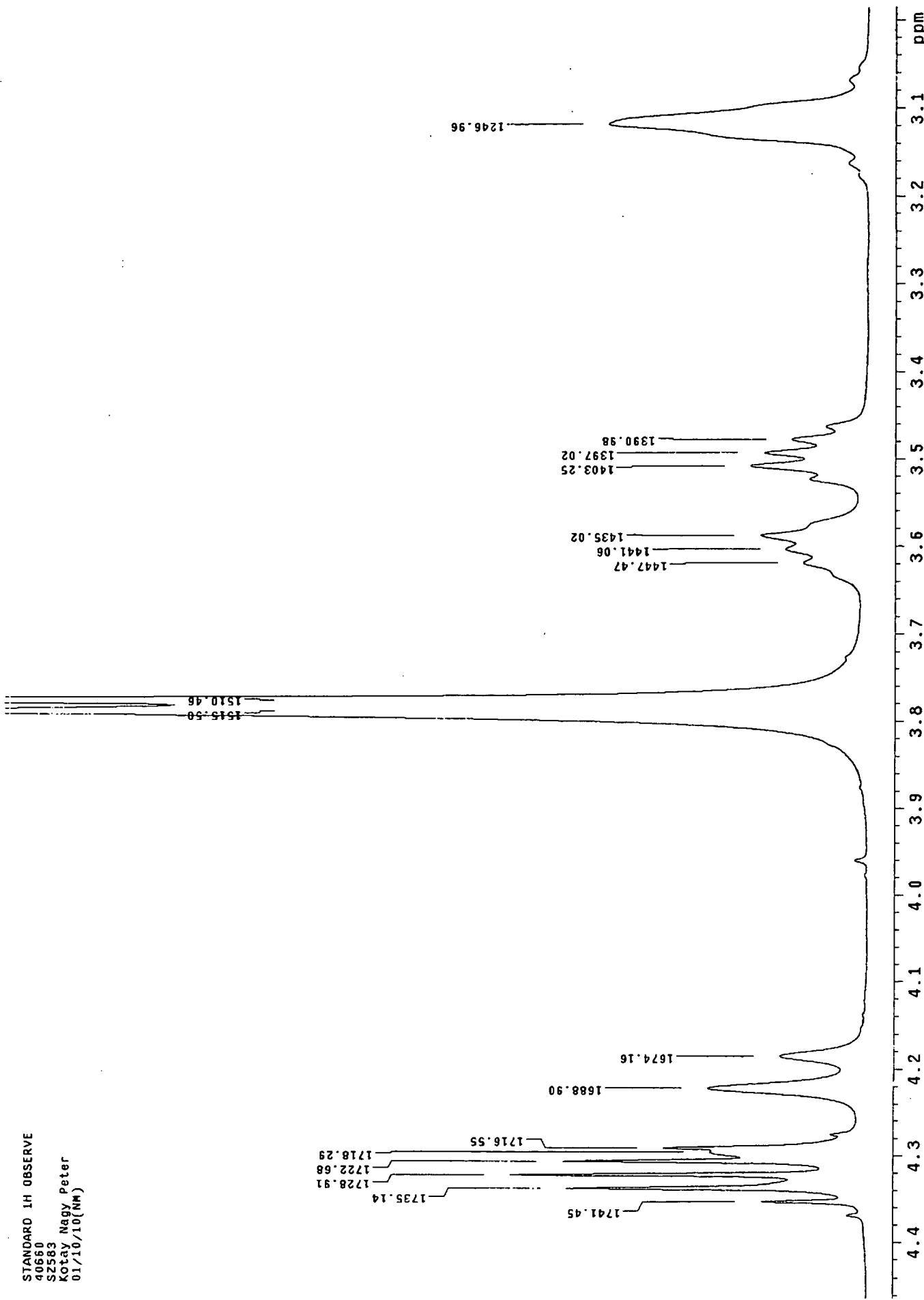
ppm

2.01 1.03  
2.04 1.01

1.94 1.01  
2.15 2.06

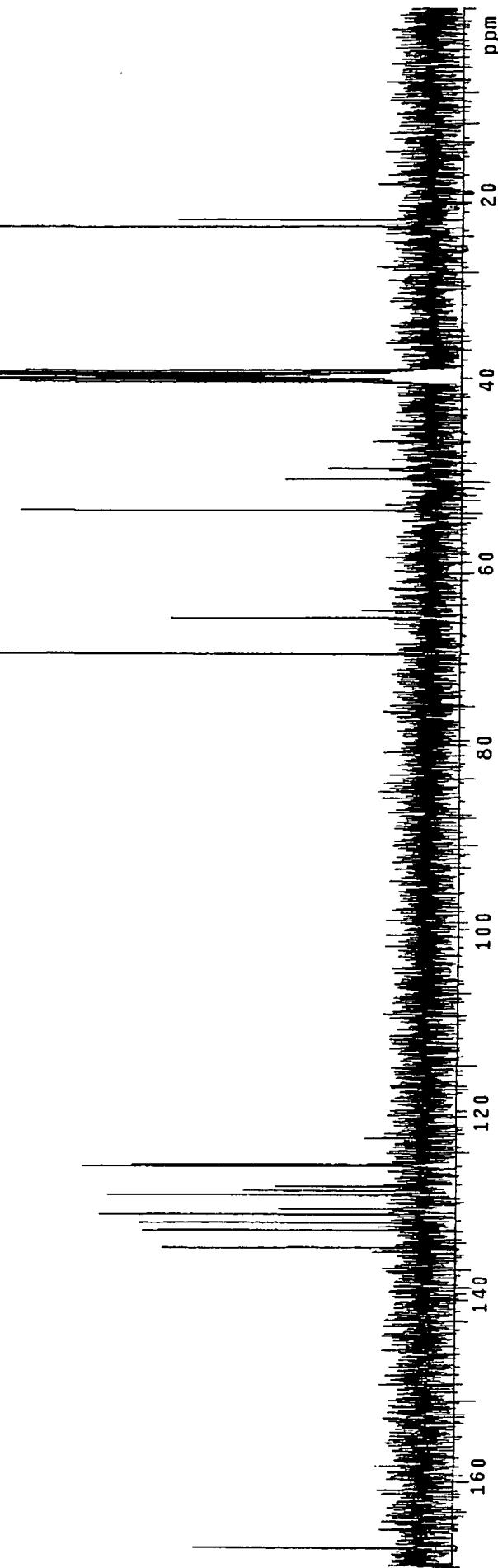
5.94  
5.94





**13C OBSERVE**  
 40660  
 S2583  
 Kotay Nagy Peter  
 01/10/10(NM)  
**Solvent: DMSO**  
**Temp. 25.0 C / 298.1 K**  
**File: 406601C**  
**INOVA 500 "1500"**  
**PULSE SEQUENCE**  
**Relax. delay 1.700 sec**  
**Pulse 41.5 degrees**  
**Acq. time 1.300 sec**  
**Width 2500.0 Hz**  
**640 repetitions**  
**OBSERVE C13, 100.5822822 MHz**  
**DECUPLE H1, 400.0108210 MHz**  
**Power 30 dB**  
**continuously on**  
**WALTZ-16 modulated**  
**DATA PROCESSING**  
**Line broadening 1.0 Hz**  
**Sq. sine bell 1.300 sec**  
**Shifted by -1.300 sec**  
**FT size 131072**  
**Total time 32 minutes**

INDEX	FREQUENCY	PPM	HEIGHT
1	16690.767	167.923	36.6
2	13573.124	134.940	42.0
3	13381.245	133.033	45.2
4	13238.466	132.210	45.7
5	13206.913	131.299	52.1
6	13147.786	130.712	23.5
7	12991.002	129.153	50.8
8	12942.555	128.671	29.0
9	12895.253	128.201	23.8
10	12872.856	125.990	54.9
11	12653.401	125.796	46.9
12	7039.311	69.983	72.7
13	6646.016	66.073	41.5
14	6564.763	65.265	10.8
15	5470.326	54.384	65.7
16	5122.044	50.922	23.1
17	5007.222	49.780	16.4
18	4056.599	40.330	65.8
19	4035.618	40.121	200.0
20	4014.637	38.912	397.1
21	3993.275	39.700	462.1
22	3972.294	39.491	394.6
23	3951.313	39.283	196.3
24	3930.333	39.074	64.9
25	2369.740	23.559	128.4
26	2291.539	22.782	40.6



0660	Kóta-Nagy Péter	MM
Z583	KBr	09/10/2001

BRUKER VECTOR22  
 Resolution: 2 cm<sup>-1</sup>  
 Number of Scans: 8

